WORK WITH A NOBLE GAS SCINTILLATION COUNTER

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UCRL-8295
Instruments Distribution

UNIVERSITY OF CALIFORNIA
Radiation Laboratory
Berkeley, California

Contract No. W-7405-eng-48

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May 21, 1958

Submitted in partial fulfillment
of the requirements for the degree of

MASTER OF SCIENCE
IN
PHYSICS

United States Naval Postgraduate School
Monterey, California

Printed for the U. S. Atomic Energy Commission

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ABSTRACT

More than 1450 runs have been made and analyzed with a noble gas scintillation counter and its associated equipment. The counter itself consists of a special photomultiplier tube looking into a scintillation chamber, and is used in conjunction with a vacuum and filling system, an output-pulse amplification system, and a 12-channel pulse-height analyzer.

Because gas contamination presented a major problem, a dynamic gas-purification system was designed and effectively employed. Adequate mixing of the binary mixtures of argon and helium was a second major problem and was solved by a specially designed diaphragm pump and by close control of the flow of the constituent gases.

Although the results obtained were most conclusive with pure argon and pure helium, sufficient work was completed on intermediate mixtures of these two gases to indicate that further investigation in this direction is appropriate. It is planned that future investigations will be made using xenon and krypton.

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INTRODUCTION

Gaseous scintillation counting essentially involves detecting the light emitted by gas molecules that have been excited or ionized by the passage of a charged particle. The first successful attempts in this direction employing the noble gases were made by Carl Muehlhause[1] in 1953, using pure argon and pure helium and detecting Pu alpha particles. His work was closely followed in the same year by Segrè and Wiegand,[2] who showed that the light output of an argon counter increased with the application of an electric field. In this case, the gas cell was actually operating as a proportional counter except that light, rather than electrons, was used to measure the event.

In their first satisfactory gas cell, Eggler and Huddleston[3] used argon at a pressure of 5 atmospheres and were able to obtain a resolution of 15% for 5.4-Mev Pu^{238} alpha particles. A plastic wave shifter was used to shift the ultraviolet light into the visible region, but its use unfortunately introduced a contaminant into the system which gradually decreased pulse heights with time. The counter reported by Robert W. Dickieson has eliminated the need for a wave shifter by using a quartz window on the scintillation chamber.[4]

It is the purpose of the report which follows to extend the experimental range of the work started by T. M. Jenkins of the Radiation Laboratory and by Dickieson.[4] Indeed, the majority of the theory, planning, procurement of equipment, and initial organization of this experiment was done by them. For all these very necessary efforts, the writer is especially and sincerely grateful.

Further, the author wishes to express his appreciation to Dr. Burton J. Moyer and Dr. Roger W. Wallace for their encouragement and supervision. Particularly, the author is grateful to T. M. Jenkins, Technician, whose interest and direct participation in most phases of the experimental work were invaluable.

This work was done in part under the auspices of the U. S. Atomic Energy Commission.

GENERAL

Basically, gaseous scintillation counting involves establishing a system whereby the light output of gas molecules that have been excited or ionized by charged particles may be detected, measured, and analyzed. Most of the efforts in this field have dealt with the noble gases, both primary and in mixtures. The major differences in these efforts have consisted of variations in methods of purification of the basic gas or gases, the geometry of the counter proper, and the extent and general arrangement of the associated systems necessary for the control of the physical constituents and for the measurement and analysis of the experimental data.

In this experiment, a dynamic gas-purification system has been employed (it is covered in detail in a subsequent section). This system keeps gas of high purity continuously available. Additionally, the scintillation chamber has a quartz window, thus eliminating the necessity for a wave shifter and hence eliminating the possibility of contamination of the gas from this source.

The original design of the chamber itself was a steel circular cylinder 7 inches deep (source to quartz end window) and 4 inches in diameter. The inner surface of this chamber was coated with aluminum oxide for good light scattering. The original geometry and surface were modified, however, and the present configuration is as shown in Fig. 1. The modified chamber is constructed of cold rolled steel and the inner surface is aluminized for good light reflection.

The entire system is constructed to be vacuumtight and can withstand up to 165 psi, although most of the data were obtained in the range from vacuum conditions up to 75 psi. A photograph of the general arrangement of the system is shown in Fig. 2 and a close-up of the scintillation-chamber stack in Fig. 3. Subsidiary systems necessary for conduct of the experiment are covered in detail in the following pages.

The various conditions of temperature and pressure--from approximately $+22^{\circ}$ C to -168° C, and from vacuum to 150 psi--were

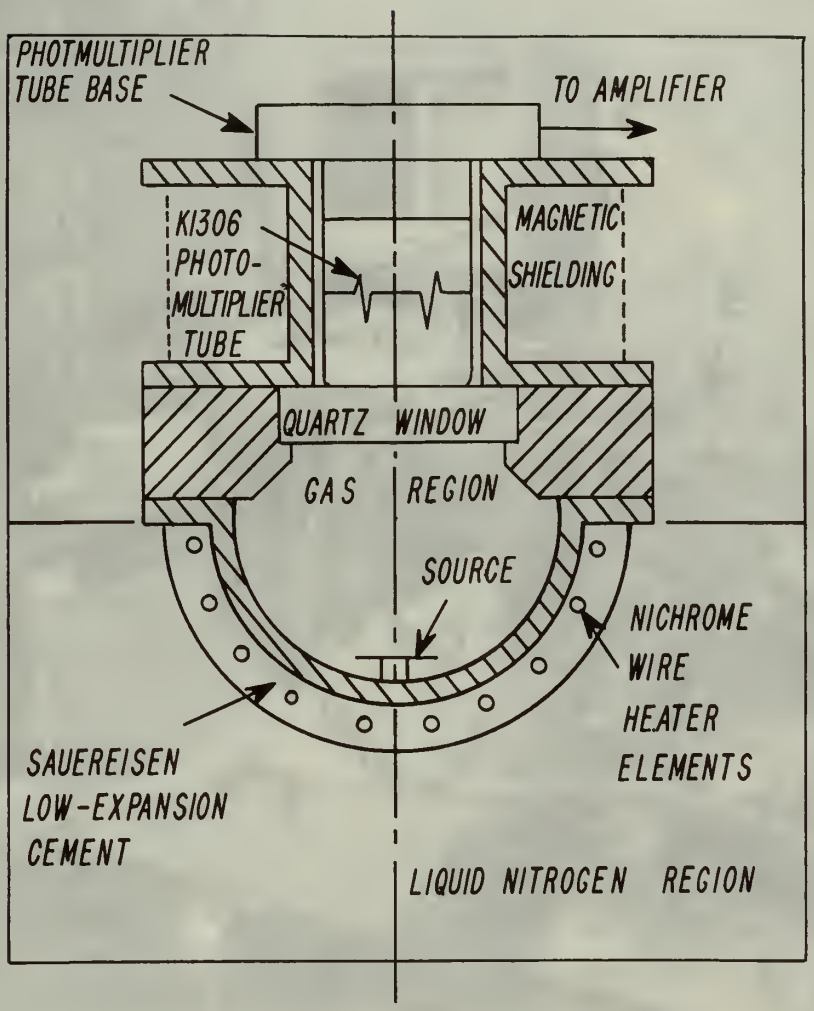

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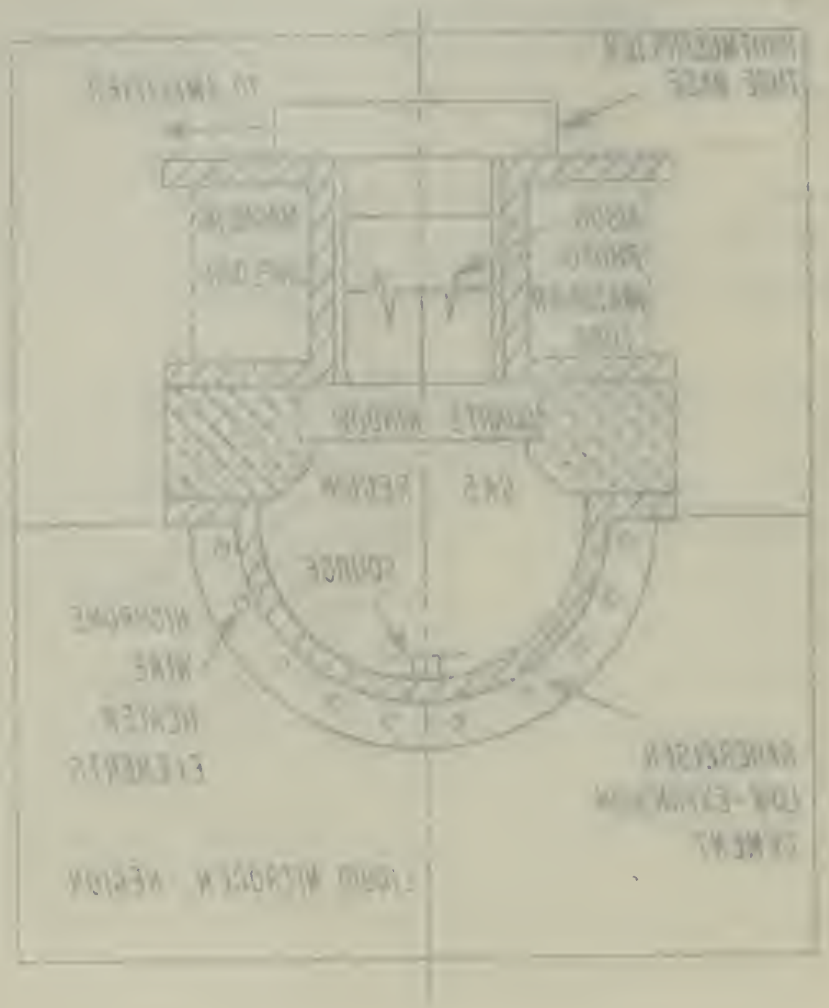
Fig. 1. Schematic diagram of scintillation chamber. Chamber is surrounded by a dewar of liquid nitrogen (not shown). Cooling for the photomultiplier tube is provided in the region of magnetic shielding.



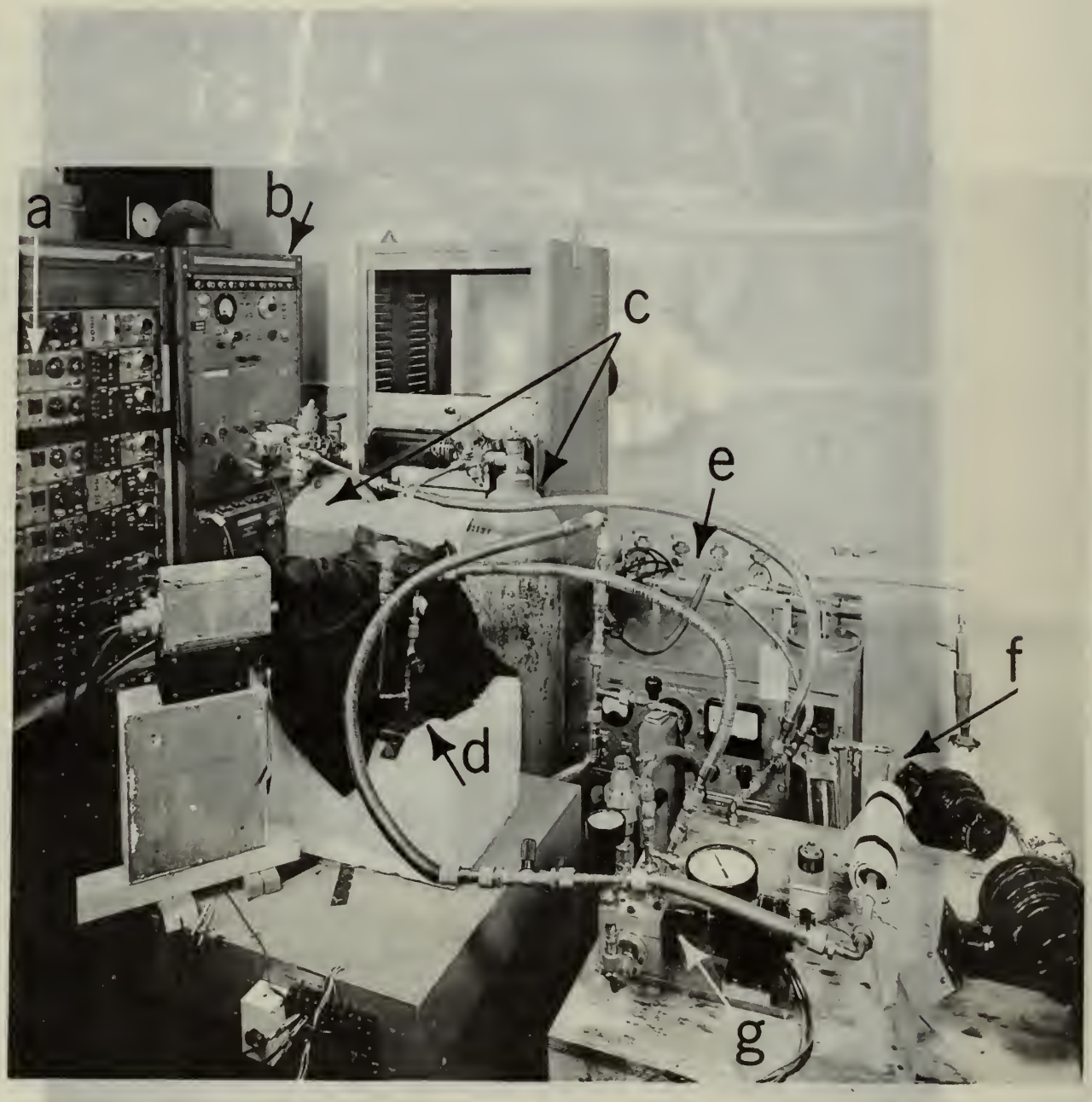


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Fig. 2. General arrangement of equipment.

a. Twelve-channel pulse-height analyzer.
b. Electronics stack (calibration pulser, linear amplifier, pulse shaper, coaxial switch, power supplies).
c. Argon and helium supply bottles.
d. Scintillation chamber stack (see Fig. 3).
e. Vacuum system.
f. Gas-purification tube.
g. Gas-circulating pump.


C
B
A
D

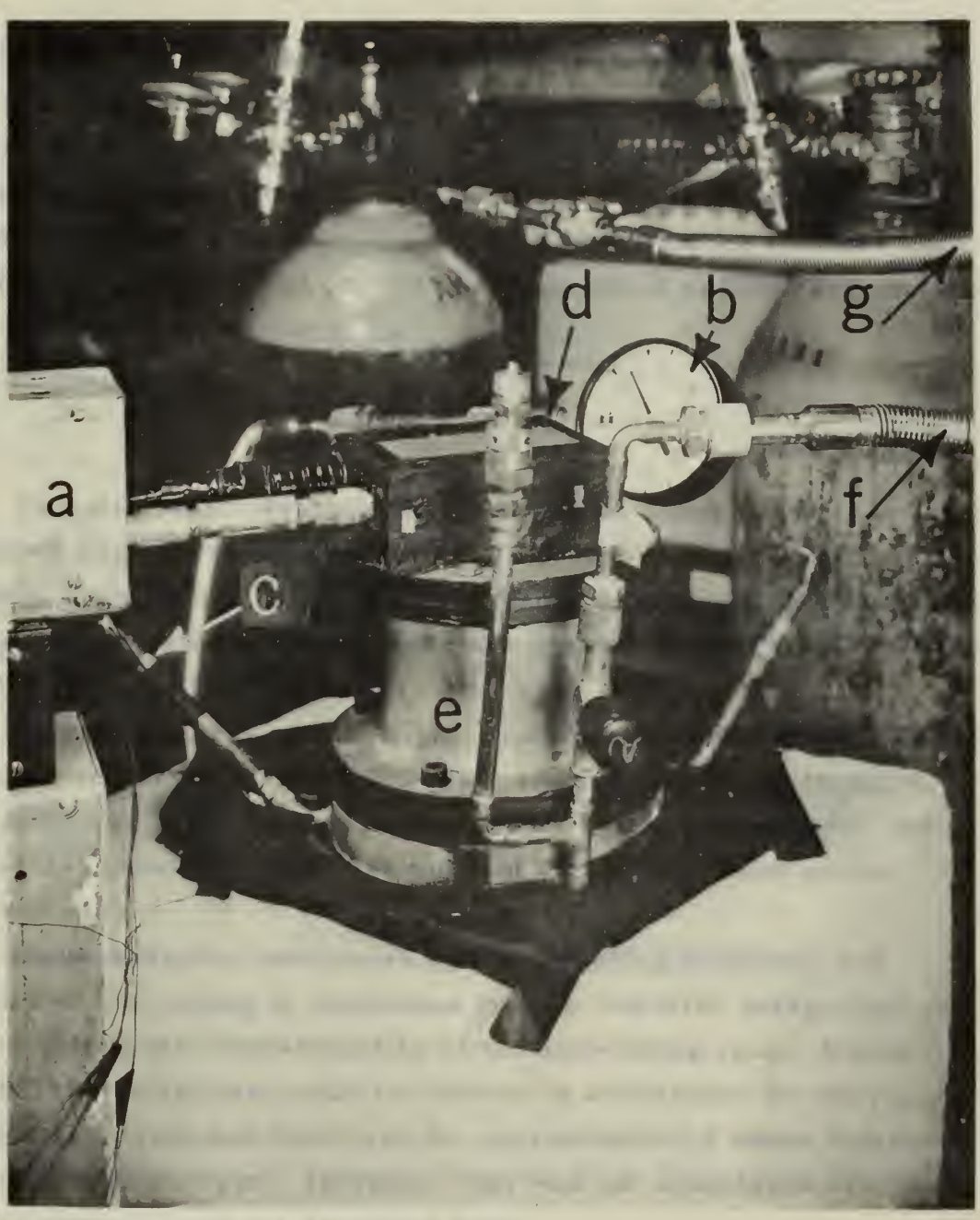

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Fig. 3. Scintillation-chamber stack.

a. Low-noise preamplifier.
b. Scintillation-chamber pressure gage.
c. Kovar seal with connections for thermocouple inside chamber.
d. Photomultiplier tube base.
e. Magnetic shielding for photomultiplier tube.
f. Line leading to gas-circulating pump.
g. Gas-filling line.

investigated both in primary argon and helium and in mixtures of these two gases. Such wide variations of temperature, pressure, and gas composition made an imposing task of gathering pertinent data. For this reason, some sections of the report that follows contain data and results covering this entire range of conditions; in other sections the treatment is limited to that part of the range which preliminary analysis shows to be of interest.

ELECTRONICS

The electronics system employed was as shown in the block diagram of Fig. 4. Circuit diagrams of the low-noise preamplifier, the Model V linear amplifier, the calibrating pulser, and the timing-pulse generator and pulse shaper are presented as Figs. 12 through 15 in the Appendix (pages 29 to 32).

As shown by Fig. 3, the signal from the photomultiplier tube is sent to the low-noise preamplifier (Fig. 12) and then to the Model V linear amplifier (Fig. 13). It is then shaped by the pulse shaper and timing-pulse generator (Fig. 14) and sent to the 12-channel pulse-height analyzer.

Photomultiplier-tube noise was a continuing problem, and necessitated the making of continuous runs to establish background corrections needed for standardization of the data-taking runs. It was found that the noise level could be reduced to a minimum by cooling the photomultiplier tube and tube base for approximately 2 hours beforehand with liquid nitrogen. However, this was not considered necessary for most runs and was not generally done.

GAS-PURIFICATION SYSTEM

In their initial planning of this project, Dickieson and Jenkins decided upon a dynamic gas-purification system.[4] This was deemed necessary because of the quenching of pulse heights due to contaminants

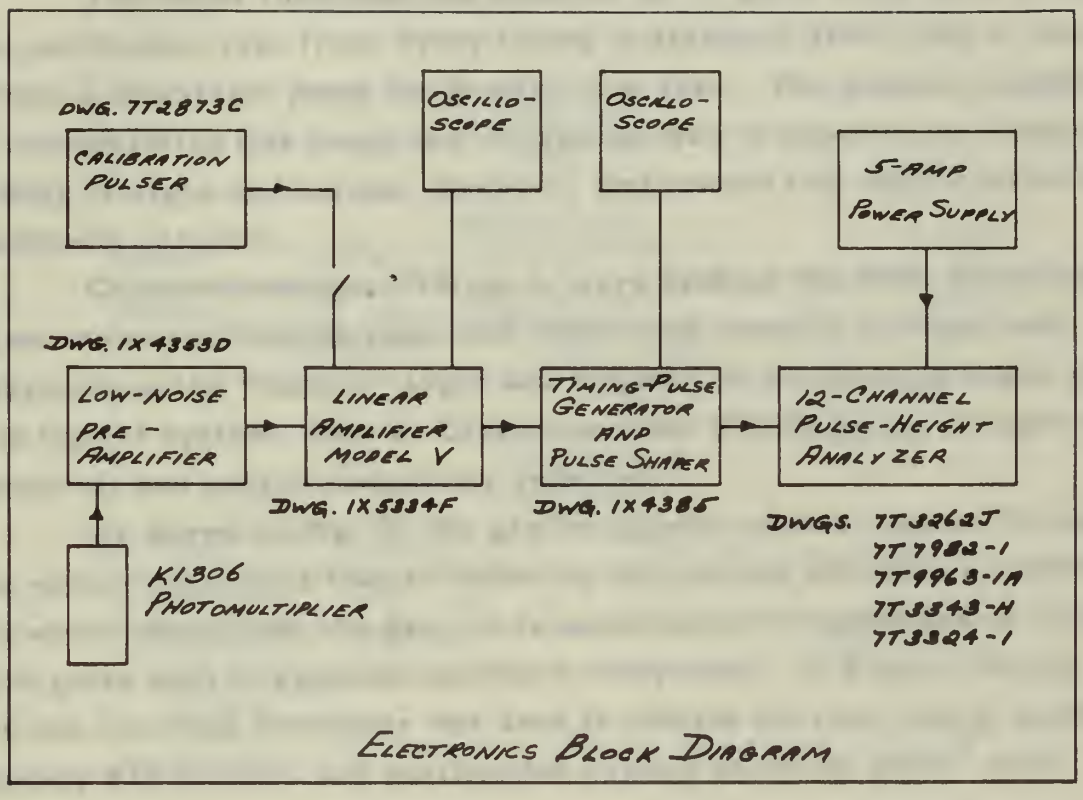

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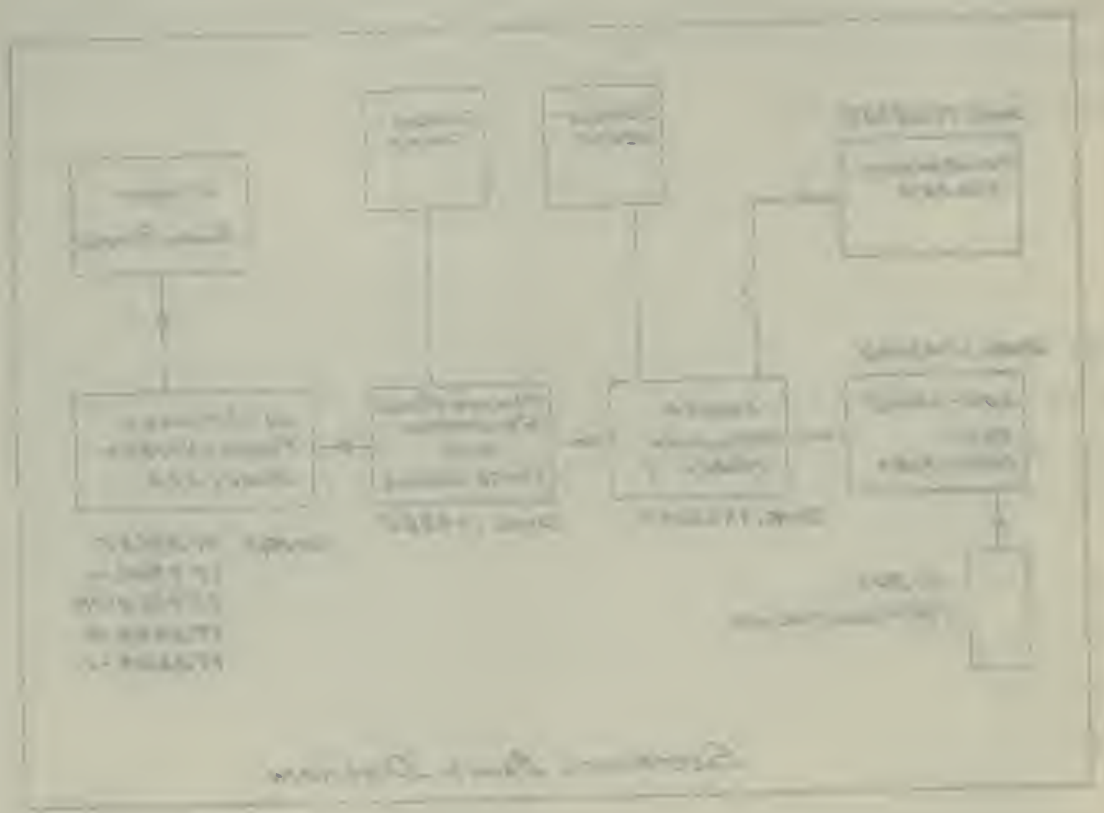
Fig. 4. Block diagram of electronic components associated with noble gas scintillator. Circuit diagrams of the individual components are in the appendix. K1306 photomultiplier is a special quartz-window tube which eliminated the use of a wave shifter.



in the basic gas system, and permits a final gas of high purity to be continuously available. Although initial efforts in this direction were primarily confined to purifying argon, this system is also suitable for purification of helium. A flow diagram of the system is shown in Fig. 5.

The major modifications made to the original setup were to change the purification tube from Vycor tubing to stainless steel, and to incorporate a diaphragm pump for greater flow rate. The primary purpose of incorporating this pump into the system was to expedite the complete mixing of argon and helium, however, and toward this end its effect was extremely valuable.

Calcium turnings, 97% pure, were used as the basic purifying agent in the purification tube, and effectively removed nitrogen and oxygen from the "impure" argon and helium. In his work on argon with this type of system, Dale S. Gibbs found that 99.93% of the nitrogen was removed, and oxygen completely removed.[5]

As shown in Fig. 5, the gas being purified first passes through the water trap. This trap is cooled by dry ice and effectively removes all water vapor from the gas. It is constructed of copper and is filled with glass wool to preclude moisture carry-over. A Fisher-Porter tri-flat low-flow Flowrator was used to control the flow rate at approximately 830 cc/min, and purification results shown by Gibbs[5] were obtained.

The purification tube itself was surrounded by 1/16-inch sheet asbestos for insulation, was heated by a nichrome wire heating element, and was maintained at approximately 650° C by a Gardsman Model J temperature controller in conjunction with an iron-constantan thermocouple. Under these conditions of temperature and flow rate, each charge of calcium turnings can be used to purify approximately 1300 liters of gas before it becomes calcium oxide or calcium nitride.

From the purification tube, the gas goes to the diaphragm pump and then to the scintillation chamber. As can be seen from Fig. 5, provision is made for recirculation of the gas where such recirculation

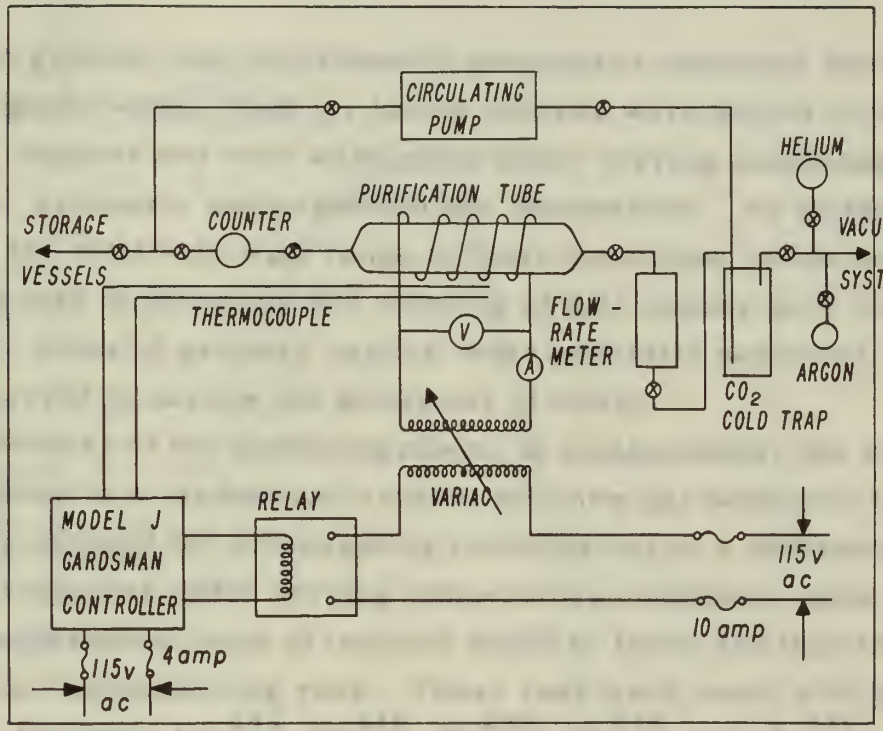
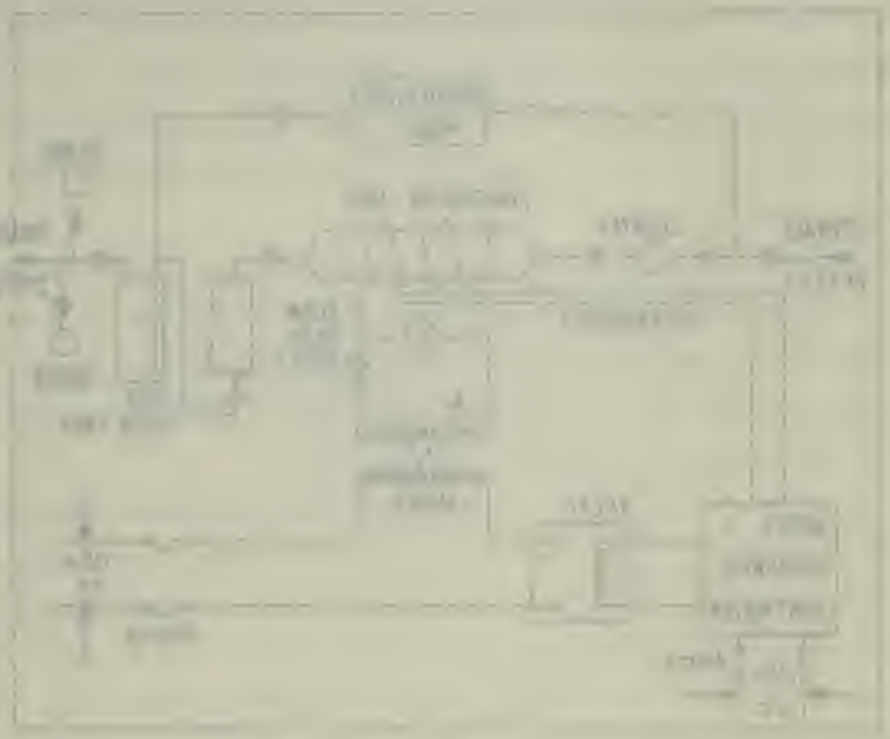


Fig. 5. Flow diagram of the gas-purification system. The Gardsman controller maintained the purification tube at approximately 650° C. The cold trap was for removal of the water vapor from the gas.



and the higher-purity gas it permits are necessary or desirable. This entire system is connected to a standard vacuum system consisting of a forepump, a diffusion pump, and associated equipment.

EXPERIMENTAL PROCEDURES

In general, the experimental procedures employed were basic and straightforward. That is, known sources were placed in the scintillation chamber and runs were made under varying conditions of temperature, pressure, and argon-helium composition. As stated previously, the relatively wide range of these conditions inside the chamber made the task of gathering and reducing of data appear quite formidable. However, areas of primary interest were generally narrowed, and this in turn served to narrow the directions of effort.

Because of the quenching effects of contaminants, the system was pumped down to a vacuum and refilled with new gas whenever the setup was being changed for investigating scintillations at a different pressure. For investigations under varying temperature conditions, data over the entire temperature range of interest would be taken and then the gas changed for substantiating runs. These runs were made with numerous sources, including Cm^{244}, Po^{210}, Ra^{226}, Np^{237}, and Pu^{239}. The great majority was conducted with the Cm^{244} source, however, and the curves showing the results (Figs. 6 through 9) are all based on its use. The Cm^{244} was electroplated on an aluminum disc 0.8 inch in diameter and 5 mils thick, and placed at the bottom of the chamber as shown in Fig. 1, approximately 1 inch from the quartz window of the chamber.

The original data obtained for argon-helium mixtures were inconclusive, primarily because of inconsistent degrees of mixing of these two gases. For almost all mixtures it was found that excessively long periods of time were required for complete mixing to take place. For this reason, a diaphragm pump was designed and built for this system. Its use greatly expedited the mixing of argon and helium and materially reduced the time required for scintillation conditions to stabilize.

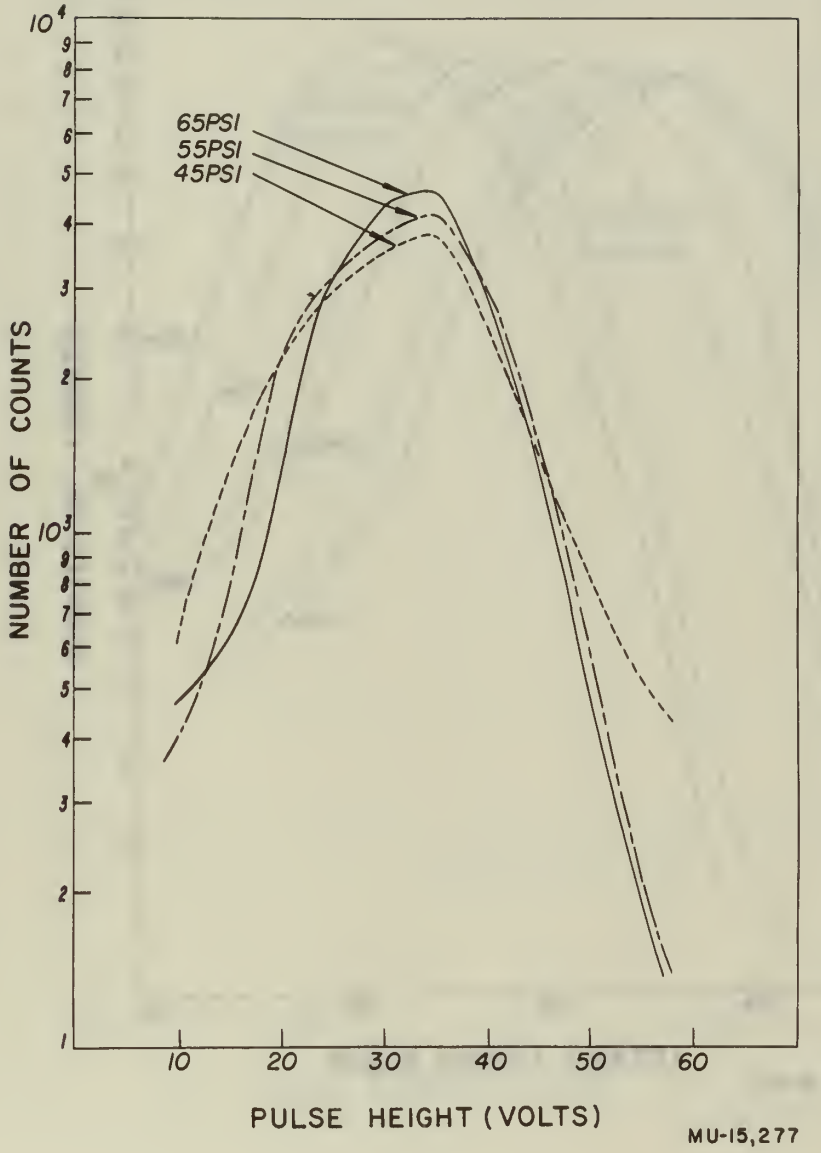


Fig. 6. Pulse height spectra for increasing pressure in argon at 22° C. Cm^{244} source.

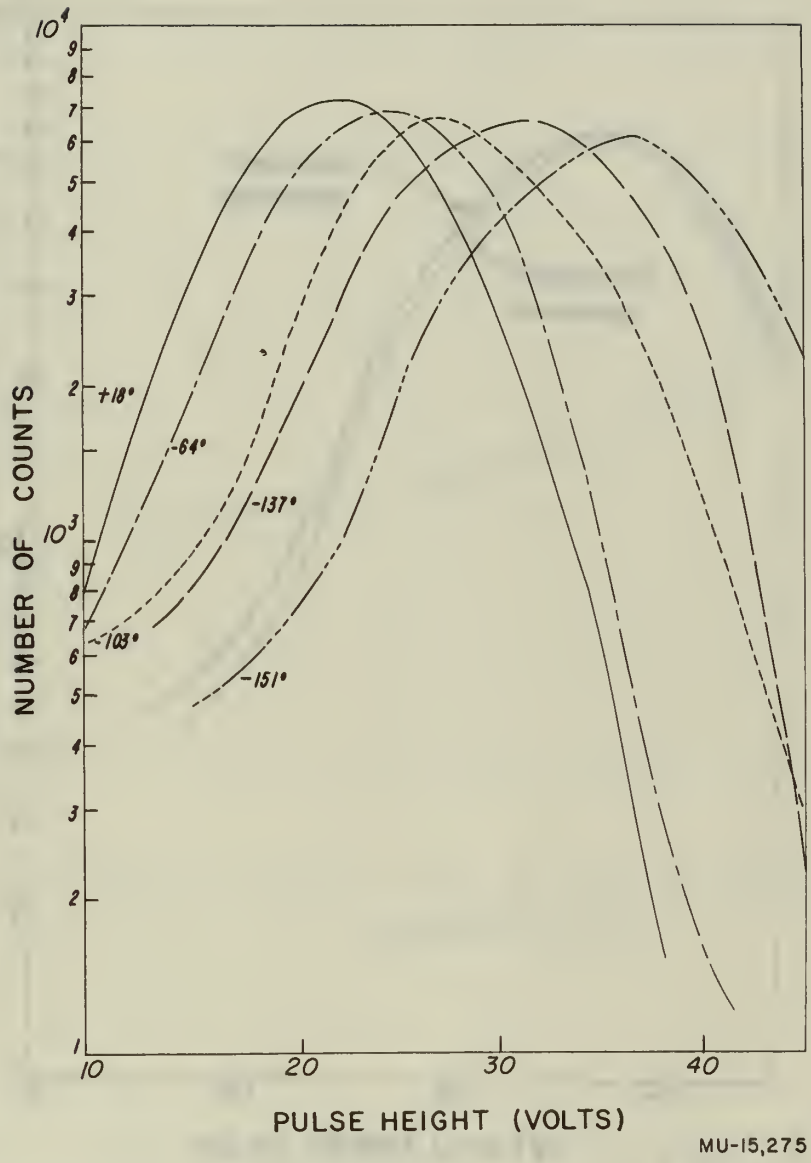


Fig. 7. Pulse height spectra for decreasing temperature in argon at 75 psi. Cm^{244} source.

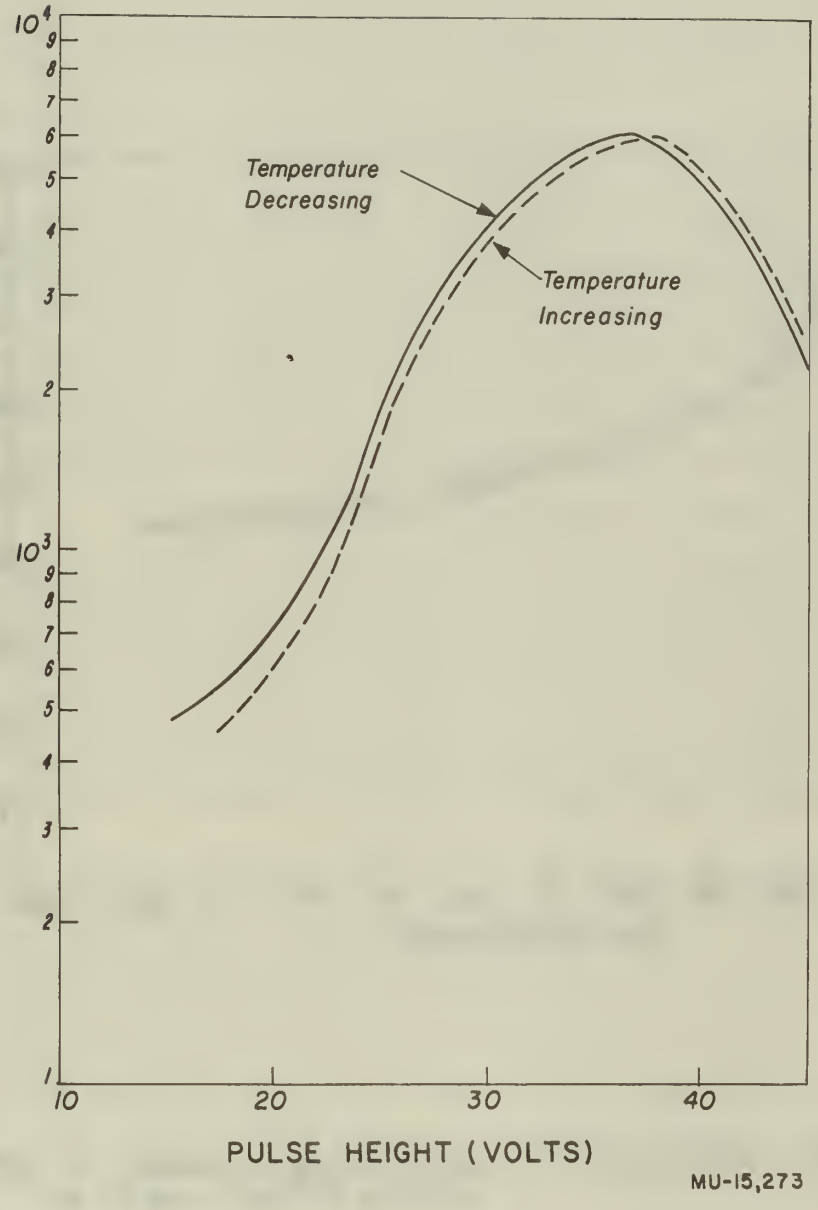


Fig. 8. Comparison of pulse height spectra in argon at 75 psi. Both curves at -151° C.

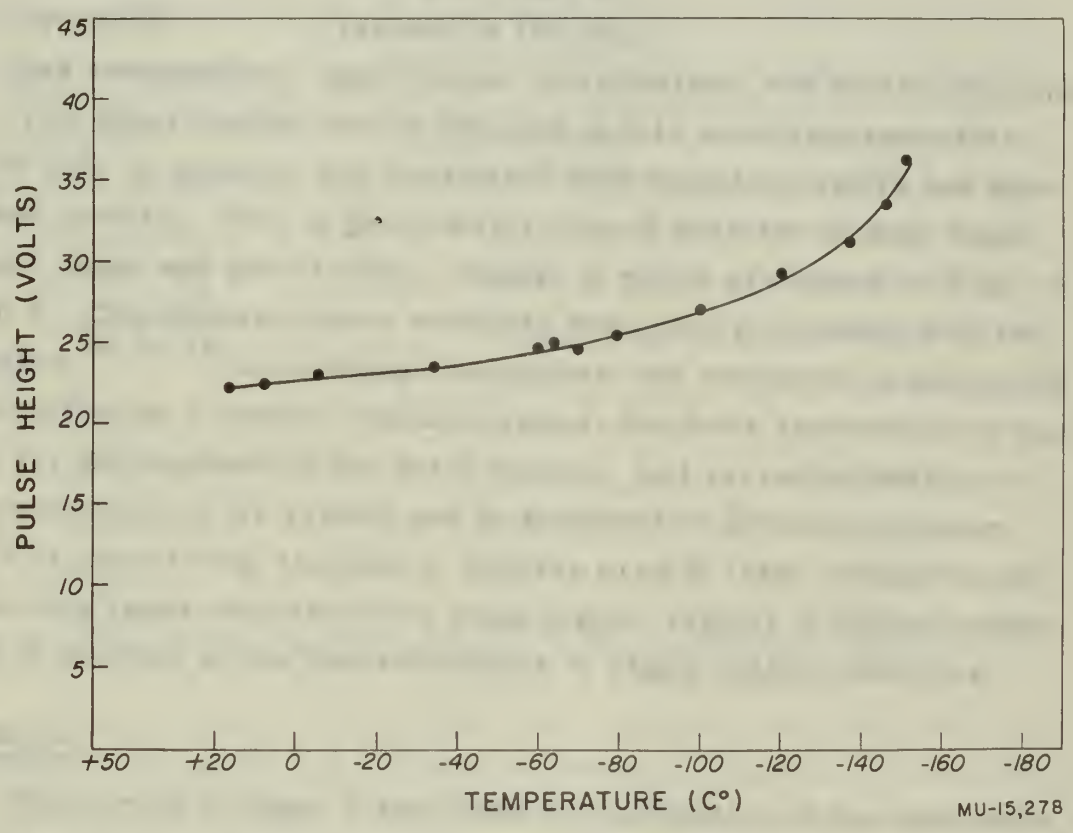
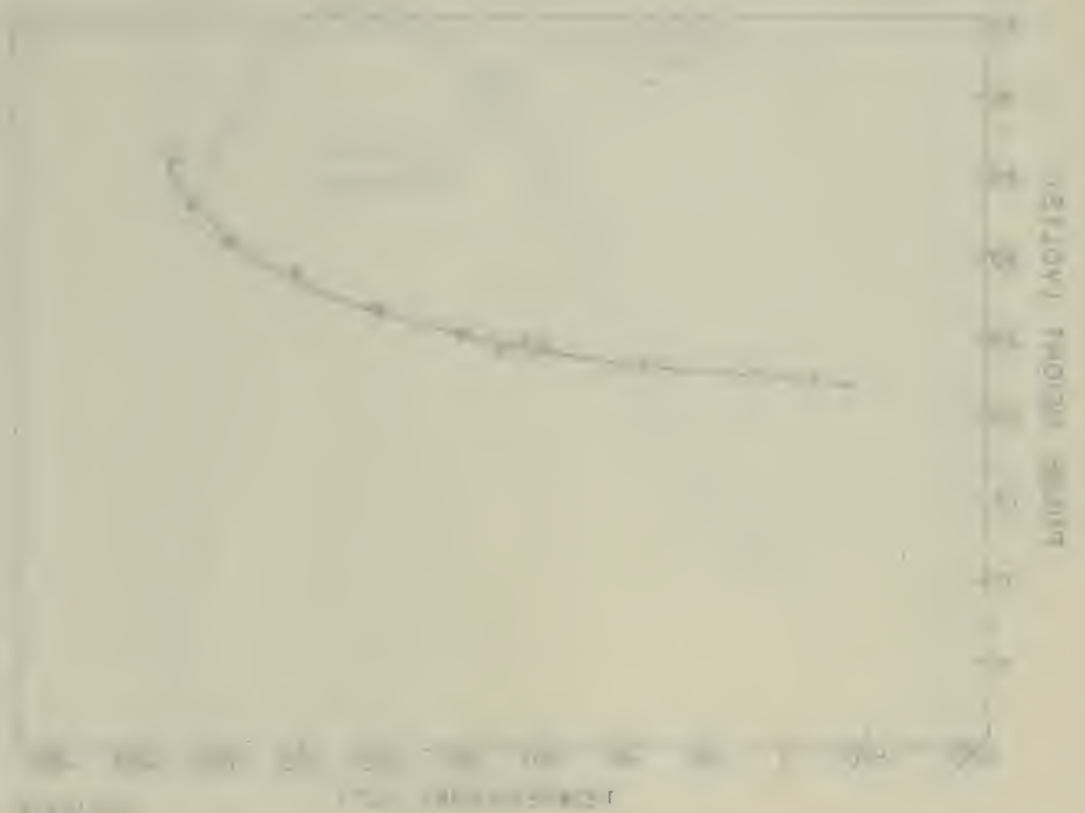


Fig. 9. Maximum pulse height vs. temperature. Pure argon at 75 psi. Cm^{244} source.



RESULTS

General

Analysis was made of more than 1450 runs conducted within the following range of conditions in the scintillation chamber:

temperature: $+22^{\circ}$ C to -168° C;

pressure: vacuum to 150 psi;

gas composition: pure argon, pure helium, and binary mixtures.

The experimental curves obtained in this work represent this analysis and, in general, are consistent with expected results and with published results. This is particularly true of analyses of runs made with pure argon and pure helium, results of which are shown in Figs. 6 through 9. Considerably more work has been done previously with the pure gases,[8, 9, 10] and although much time was consumed in analyzing their scintillation response characteristics, the work reported here was largely for development of the basic system, and served primarily to aid in refinement of the system and to substantiate previously known data. It is considered, therefore, that the area of least conclusive evidence in this report and hence the most logical subject of future investigations is analysis of the characteristics of argon-helium mixtures.

Pure Gases

The curves in Figs. 6 and 7 are representative of the work done on the pure gases, and show results obtained for pure argon. Figure 6 represents the change in relative pulse height with change in pressure at a constant temperature, and parallels the results and conclusions obtained by other workers.[7, 9] The curves show essentially that once the distance from the source to the phototube exceeds the range of the alpha particle in that gas, the increase in pulse height with increase in pressure is very gradual, but the resolution improves markedly. This effect--once a pressure is reached assuring all the alpha particles' being stopped in the gas--is probably due to the concentration of the alpha tracks in the center of the chamber as the pressure increases.[9]

Since the distance from the Cm^{244} source to the phototube in the geometry under investigation was approximately 1 inch, it can be seen from Table I that the distance from source to phototube exceeds the range of the alpha particles in argon for all the pressures represented in Fig. 6.

Table I

Ranges of 5.8-Mev alpha particles (Cm^{244}) in argon and helium

Pressure	Range in argon (in.)	Range in helium (in.)
-20 in. Hg	4.96	26.4
-15 in. Hg	3.31	17.6
-10 in. Hg	2.48	13.2
0	1.65	8.83
20 psi	0.83	4.41
25 psi	0.63	3.31
50 psi	0.38	2.04
75 psi	0.28	1.50
100 psi	0.24	1.26

The curves in Fig. 7 represent the change in relative pulse height with change in temperature at constant pressure, and the results are quickly seen to be totally different from those shown in Fig. 6. By comparison of the two sets of curves, one immediately concludes that improvement in resolution is closely related to an increase in pressure. Temperature change, on the other hand, appears to have little effect upon resolution. (In comparing Figs. 6 and 7, one should note that the voltage scales are not the same.)

It is of interest to note, in analyzing the curves in Fig. 7, that careful plotting of data obtained as temperature was decreased to $-151^{\circ}C$ showed that they varied slightly from data obtained during the return to room temperature. This variation, shown in Fig. 8, was not great, but significant enough to note, and occurred every time the procedure was

repeated. Since the direction of temperature change (i. e., increasing or decreasing) should in itself be of no significance, it must be assumed that this slight but consistent variation resulted from differing thermal characteristics of the gas and of the phototube itself. A description of the technique involved may make this clearer.

To obtain the data for Fig. 7, argon at 75 psi was introduced into the counter chamber at room temperature. Liquid nitrogen was then introduced into the volume surrounding the chamber (Fig. 1), and data were taken as the temperature in the chamber was lowered to approximately -160° C. The thermocouple used to determine the temperature inside the volume was on the centerline of the volume and approximately 1 inch from the quartz window (Fig. 1). It was necessary to carefully control the input of argon as the temperature decreased in order to maintain the pressure at 75 psi, and similarly, upon warming of the chamber as the temperature returned to $+18^{\circ}$ C, it was necessary to release argon from the chamber to maintain the pressure constant.

The general effect then, as shown in Fig. 7, is a shifting of the curve to the right as temperature decreases. This can be attributed to a combination of two effects as the temperature changes--a change in the scintillation characteristics of the argon, and a change in the response characteristics of the phototube. The variation in results with direction of temperature change is shown in Fig. 8 for -151° C and indicates that the peak of the curve as the temperature returns toward $+18^{\circ}$ C is displaced slightly to the right of the peak recorded when the temperature is decreasing. The relations between the positions shown for -151° C corresponds almost exactly to those for all temperatures between $+20^{\circ}$ C and -160° C. This variation is not completely understood, but is probably due to a difference in rate of change of temperature between the argon and the phototube. That is, since the thermocouple measuring the temperature is in the gas volume, it seems likely (from the relative positions of the two peaks in Fig. 8) that the rate of change of temperature of the phototube is less than that of the gas, and that in the case presented, the temperature of the phototube itself is somewhat lower than -151° C.

This variation may not be an important one in certain applications, but should be considered for investigations involving temperature changes and the resultant changes in gas and phototube characteristics.

Figure 9 is a compilation of the results obtained for numerous runs similar to those of Fig. 7. Because liquefaction of argon at 75 psi occurs at -167° C, and a change in scintillation characteristics would likely accompany the change in physical state, it cannot be assumed that the maximum pulse height continues upward and to the right with decreasing temperature, as the curve in Fig. 9 might infer. Investigation of scintillation characteristics of liquid argon was not the purpose of this work, therefore investigation was limited to temperatures higher than -167° C. However, this would be an appropriate extension of the studies reported herein, and it is planned to investigate these characteristics in the future.

Argon-Helium Mixtures

Although other workers have conducted investigations of mixtures of Xe in Kr, Kr in A, and Xe in He without highly promising results,[7] it was decided to investigate the scintillation characteristics of argon-helium mixtures. Control and qualitative problems immediately presented themselves, but only the problem of complete and consistent degrees of mixing of the two gases turned out to be a difficult one. The importance of complete and adequate mixing of constituent gases for valid results cannot be overemphasized, and it follows naturally that substantiation and statistical strength of results are direct functions of consistent mixing.

The problem of complete mixing was first approached by allowing the constituent gases to stand in the scintillation chamber until such time as scintillation response stabilized. This proved to be an impractical length of time in almost all cases, however, and a diaphragm pump designed and built by Leo Leidy and T. M. Jenkins of the Radiation Laboratory was incorporated into the system (Fig. 4). The use of this pump permitted the mixing time of the gases to be cut down considerably and facilitated the making of many runs in periods of time that had

previously permitted very few to be conducted. In spite of this improvement in technique, the total number of valid mixture runs is considered to be inadequate for conclusive results because a great many of the early mixture runs made were invalidated as experience and operating time indicated that they had been in the "shady area" of incomplete mixing. Consequently, it should be pointed out that the mixture data originating in this report suffer from this numerical inadequacy, and need statistical strengthening before valid conclusions may be drawn.

Figure 10 is the basic representation of characteristics of argon-helium mixtures, and may be compared with the characteristics of other binary mixtures in Fig. 11.[6] The direction of mixture change indicated by the arrows on Fig. 10 is important in analyzing this curve, and indicate two regions that are not understood. That is, in Regions A and B of the curve in Fig. 10, the peak pulse-height values recorded in this investigation consistently depended entirely upon which gas was introduced first. As can be seen by the curve, when helium is being added to pure argon (solid curve), the peak pulse height in Region A was consistently higher than the value recorded in this region when the mixture was approached from pure helium (dashed curve). Conversely, in Region B, the value recorded when the mixture was approached from pure argon was consistently lower than that recorded when the mixture was approached from pure helium.

The procedure used in obtaining values for the solid line of Fig. 10 was to introduce into the evacuated counter chamber the amount of argon required for the desired ratio in a 40-psi mixture. Then helium would be introduced to complete the mixture, and the mixture circulated by the diaphragm pump. With a system volume of approximately 250 cc, a flow rate of 900 cc/min was used to expedite mixing. The procedure employed to obtain the dotted line was to reverse the process above and introduce the helium first, then proceed to obtain a 40-psi mixture in the ratio desired. This immediately leads one to suspect complete mixing as the solution to these apparent inconsistencies, but extremely long periods of mixing time made no significant change in results obtained after scintillation conditions appeared to stabilize.

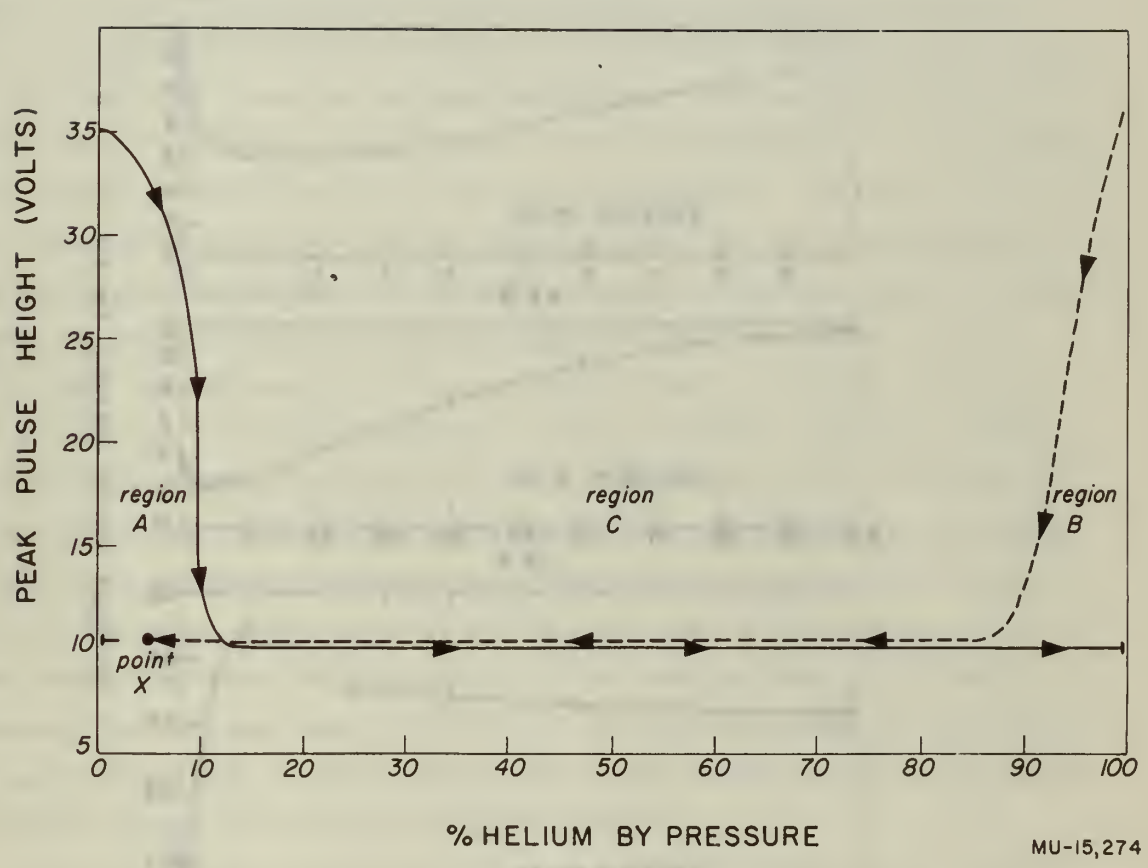
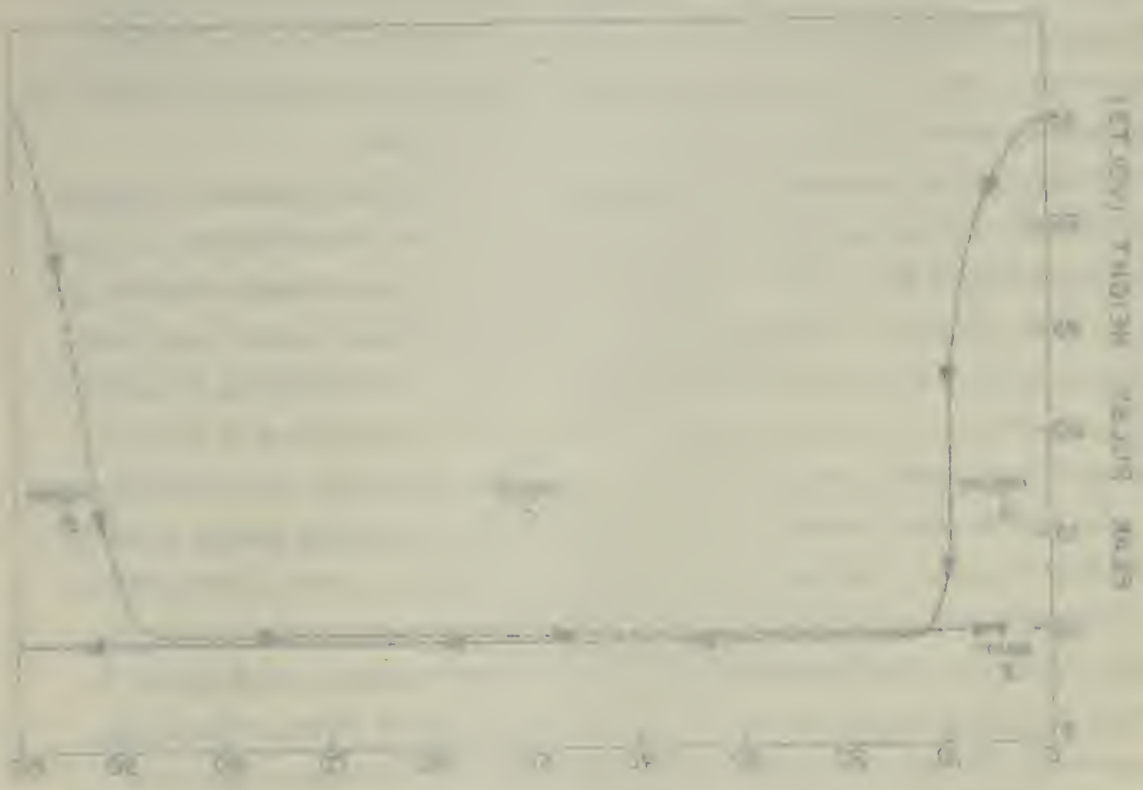


Fig. 10. Relative pulse heights, A-He mixtures. Cm^{244} source. 40 psi total pressure.



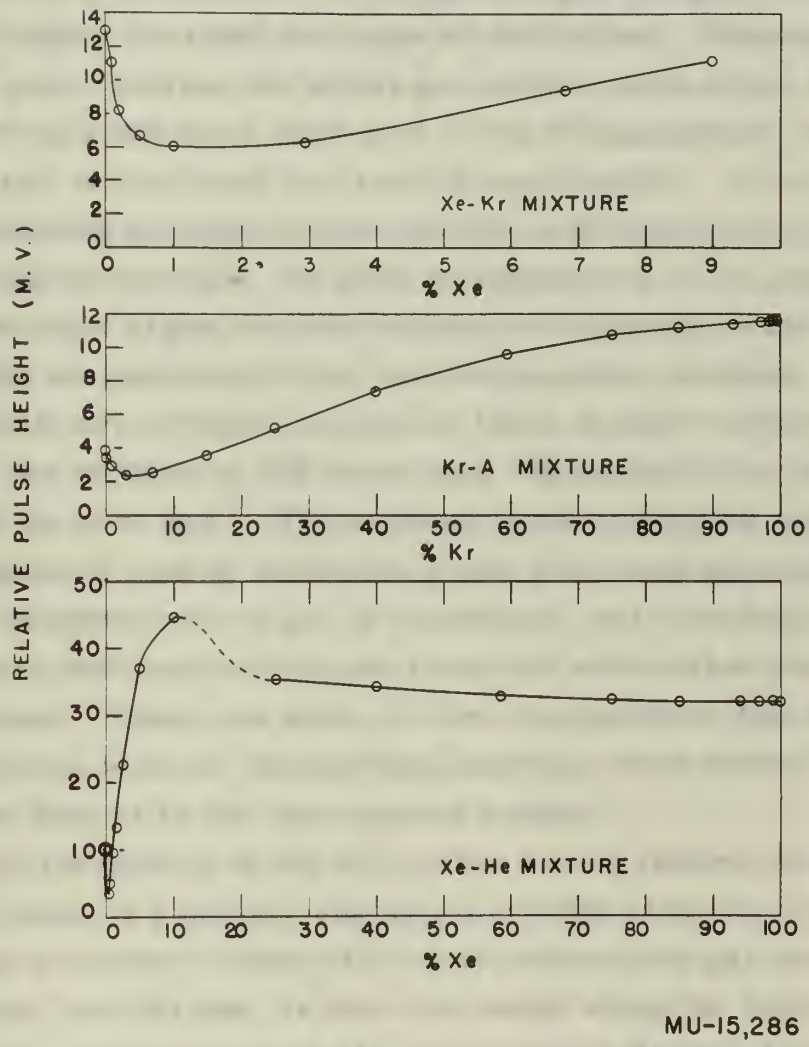


Fig. 11. Plots of relative pulse heights for xenon-krypton, krypton-argon, and xenon-helium mixtures.

The conclusion at which one arrives (after it must be assumed that complete mixing has taken place) is that a physical reaction is taking place in the evacuated counter chamber upon introduction of a gas. This reaction, not known at the moment, may vary with gas being introduced, or may be fairly independent of the specific gas being introduced and hence be roughly the same for argon as for helium. Whether this reaction takes place between the initial gas and the walls of the chamber, or between the gas and some other part of the filling system, is not known, but the result is consistent and readily reproducible. From the point of view of obtaining an argon-helium mixture with improved scintillation-response characteristics, the problem appears to be an academic one, because the pure argon and pure helium each appears to have better scintillation characteristics than any intermediate mixture. To this point, results are virtually parallel to those of other workers, which show only the mixture of 10% xenon with 90% helium to be an improvement upon the pure gas.[6] The problem is an interesting one, however, from the point of view of determining just what does take place in the evacuated chamber when a gas is introduced, and what happens when argon enters that may be different from that which takes place when helium enters. From this point of view, this problem and its solution may well prove to be of considerable practical value toward refinement of systems similar to the one reported herein.

It is the opinion of the writer that a start towards this solution is (a) that there is a definite absorption into the aluminized walls of the chamber of a certain number of the molecules of the gas introduced into the chamber, and (b) that, in fact, the points along the lower of the two lines in both problem regions (the dotted line in Region A and the solid line in Region B) should actually be in Region C.

Another way of making this statement (presently theoretical) is to consider a given point in Region A--say, that point marked "X" (5% He–95% A) on the dotted curve, the curve of values plotted when helium is the gas introduced first into the evacuated chamber. It is submitted that when the helium is introduced into the evacuated

chamber, some of the molecules are adsorbed by the walls and are not included in those that indicate 2 psi of helium (5% of 40 psi); then when the argon enters the chamber and the circulation of gas to facilitate mixing commences, certain of the adsorbed molecules are displaced by argon molecules and re-enter the gas volume proper. This would have the effect of increasing the percentage of helium and moving point "X' to the right. If moved sufficiently far in that direction, the result obtained would then be a consistent one, not inconsistent as it presently seems.

Similar analysis of the results obtained for Region B would serve to move those points on the lower line in that region to the left, and hence make these values appear more reasonable. Natural extension of this analysis leads to the conclusion (conjecture at this writing) that the final shape of the curve in Fig. 10 would be one continuous line of the general shape presently shown, except that there would not be the lower lines in Regions A and B. The problem associated with Fig. 10, then, appears to resolve itself into one of quality control--i. e., an accurate determination of the final argon-helium mixtures in Regions A and B. Solution of this quality-control problem may not, in fact, resolve the inconsistencies now associated with these regions, but this is the direction of effort now planned in the statistical refinement of the curve in Fig. 10.

Much work has been done and is continuing on the scintillation properties of the pure noble gases. However, it appears to the writer, notwithstanding the generally unpromising results of work with mixtures of gases heretofore, that reduction of data from this mixture work has presented interesting problems similar to that posed in Fig. 10, and that continuing investigation of mixtures is accordingly justified. Three problems associated with this work immediately present themselves and can be stated broadly as follows:

(1) finding a method of accurate determination of the ratio of gases;

(2) a more thorough investigation of the properties of accurately defined argon-helium mixtures, particularly in the range

near pure argon and pure helium (Regions A and B in Fig. 10);

(3) extension of this basic work into the other physical states.

Although the immediate direction of effort is toward number (1) above, it is hoped that all three of the broad areas of interest stated can be investigated; present plans now provide for such investigation.

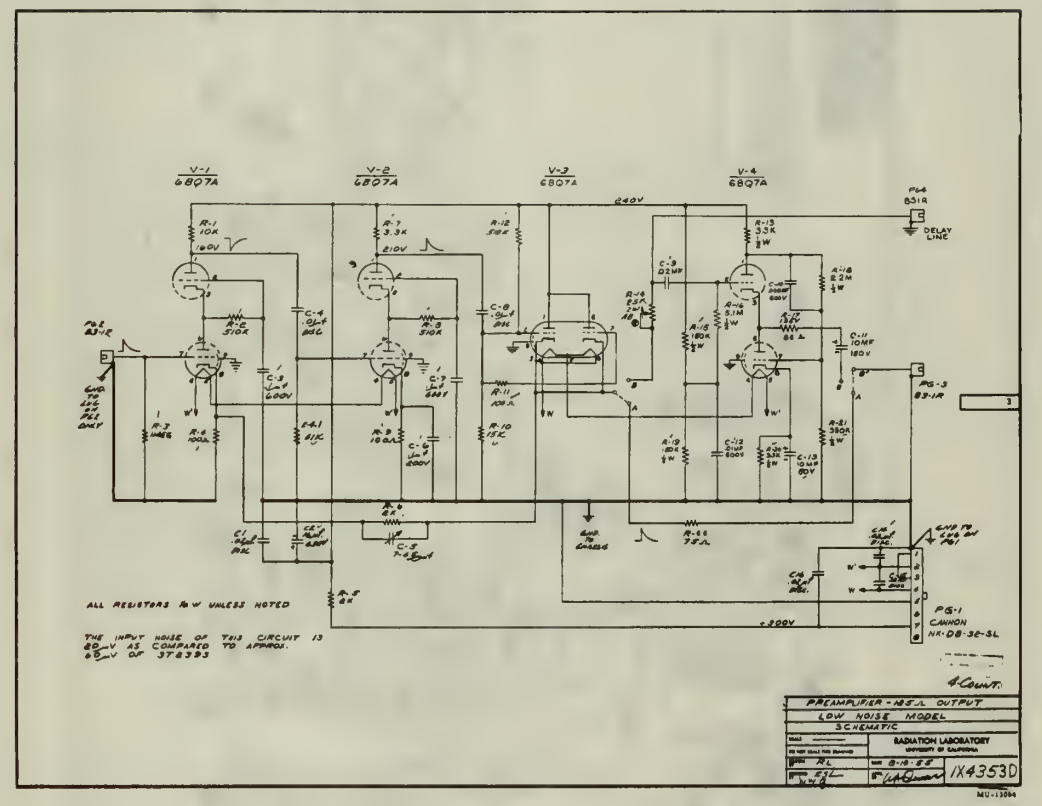
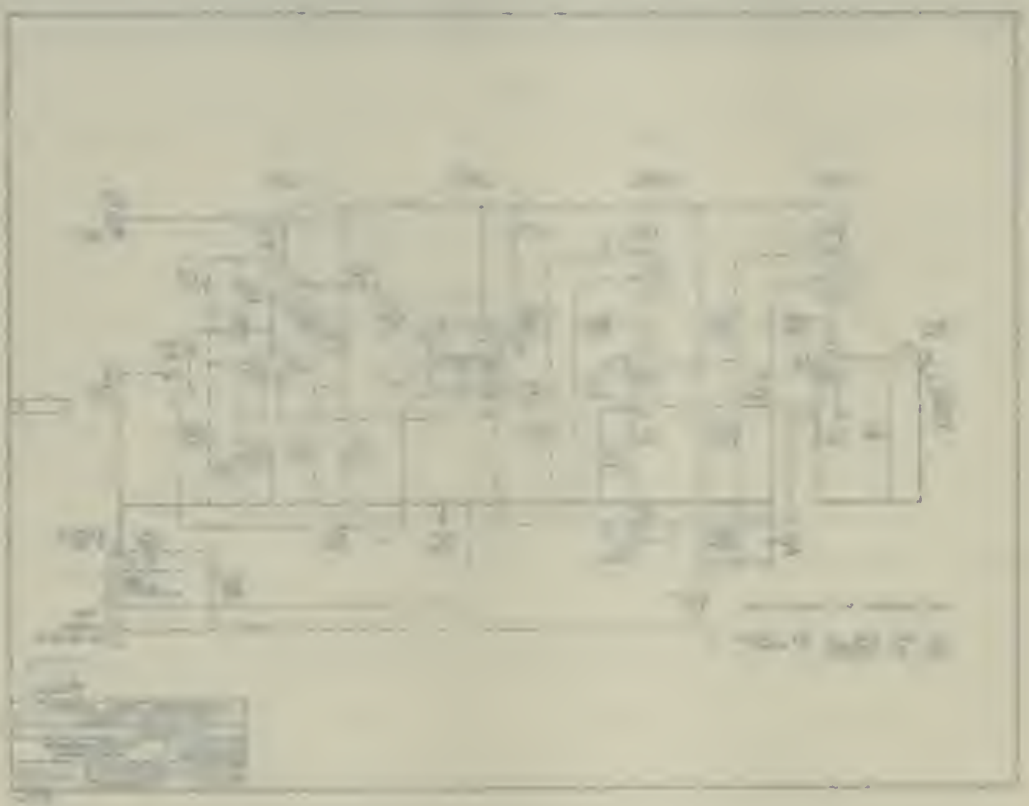


Fig. 12. Low-noise-model preamplifier (125 Ω output).



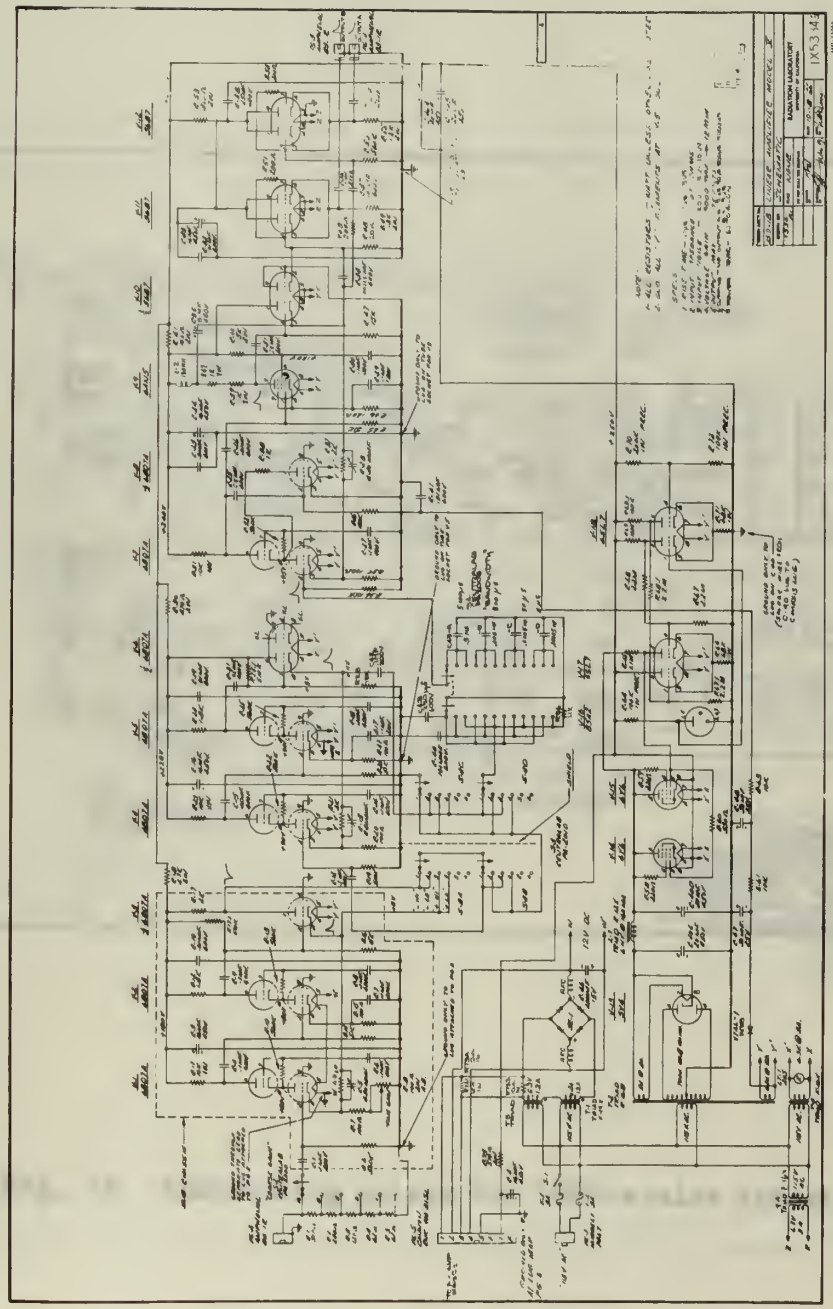


Fig. 13. Linear amplifier, Model V.

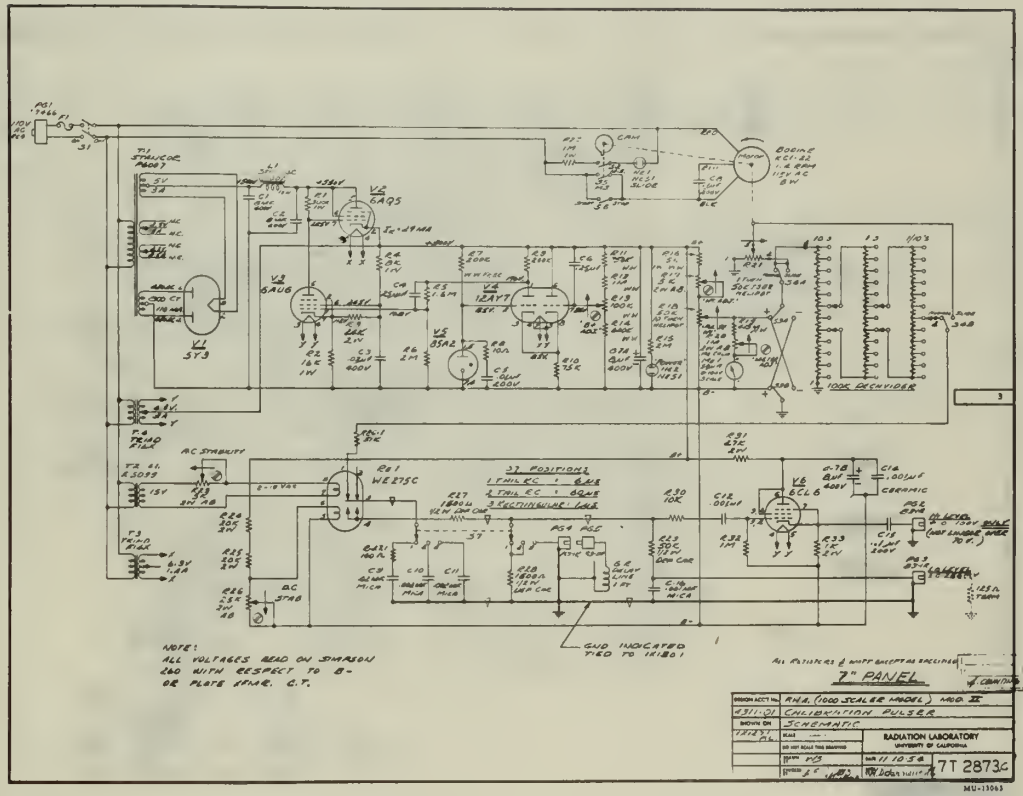


Fig. 14. Calibrating pulser for 1000-scaler Model II.

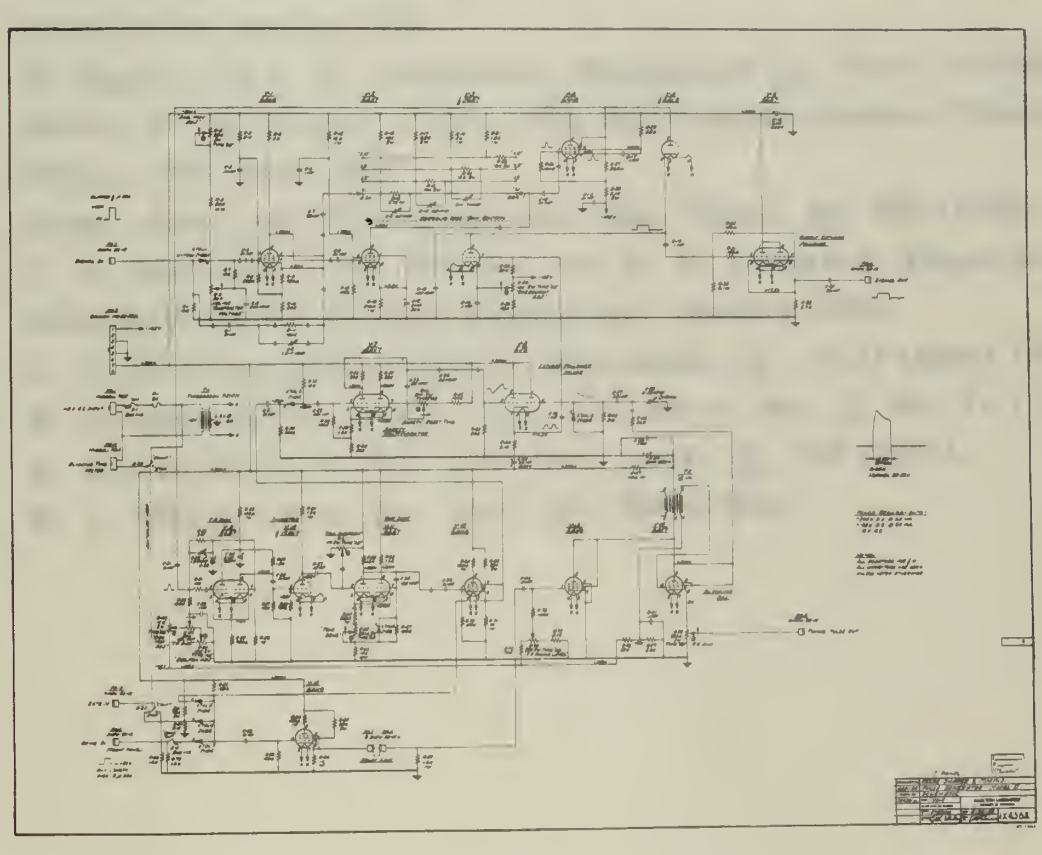


Fig. 15. Pulse shaper and timing-pulse generator, Model II.

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